BOOK TWO

RCRA Facility Investigation Workplan

RCRA Corrective Action
Steubenville East Coke Plant
Follansbee, West Virginia

1954

1966

05 November 1999
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PREPARED FOR

Wheeling Pittsburgh
STEEL CORPORATION

MICROSEEPS, INC.
LABORATORY
QUALITY ASSURANCE MANUAL

Controlled Copy No. _____

Signature of Final Approval:

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Laboratory Director

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Technical Director

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1.0 INTRODUCTION

Microseeps, Inc. (Microseeps) is committed to reliability and excellence in analysis. The laboratory provides analytical services to assist clients in complying with major environmental regulations. To achieve our goal, all data must be properly documented, legally defensible, and supported by statistically defined and verifiable confidence limits. Falsification of data under any circumstance is unacceptable and is grounds for termination. Microseeps uses EPA-approved methodologies such as those found in Standard Methods, SW-846, and ASTM whenever methods are available. If an approved method has not been specified by the EPA, Microseeps will select an industry recognized and validated method for use.

The purpose of this Quality Assurance (QA) Manual is to define the minimally acceptable standards under which all laboratory operations will be performed. As supplemented by Standard Operating Procedures (SOPs) and project specific plans, the QA Manual describes the laboratory's objectives, organization, and operating philosophy.

2.0 OBJECTIVES

Microseeps's Quality Assurance Program has been developed to ensure sample and data integrity. Tò attain Microseeps's goal of producing reliable and verifiable results, the program will address the following:

1. Detailed and documented training programs for all chemists and technicians to insure that each employee is thoroughly familiar with the methods, procedures, and record keeping pertaining to their assignments.

2. Processes for review and validation of analytical data produced at the laboratory to assure that all data are within the guidelines of this manual and associated SOPs.

3. Detection and resolution of problems through systematic procedures.

4. Documentation procedures necessary to produce legally defensible data.

3.0 ORGANIZATION AND RESPONSIBILITY

Microseeps is a small business as defined by Standard Industrial Classification 8734. Since Microseeps is a small business that must compete in a marketplace characterized by merger and consolidation, it is even more important to have an efficient organization with well-defined responsibilities. The purpose of this section is to describe the organization and to detail the responsibilities.

3.1 Microseeps Organization

Microseeps is organized as illustrated in Figure 3-1. Responsibility and dedication to accepted laboratory practices and procedures begin at the top. It is the duty of the President to assure that the framework is in place to provide the guidelines, it is the duty of the senior laboratory staff to implement the policies and procedures and to see that they drive the activities of the lab.

3.2 Responsibilities

SENIOR VICE PRESIDENT - The Senior Vice President works closely with the President to provide overall direction for the laboratory operation. He is responsible for identifying potential new markets and shares the responsibility for the financial integrity of the laboratory with the President. Additionally, he approves capital expenditures and evaluates current market conditions to maintain the laboratory's competitiveness.

MANAGER OF OPERATIONS - The Manager of Operations is responsible for implementing laboratory operations policies and procedures. He supervises the daily operations of each area of the laboratory. He is also responsible for overseeing the routine expenditures and for maintaining the laboratory's budget.

VICE PRESIDENT – CUSTOMER SERVICES - The Vice President of Customer Services provides client contact to determine project requirements and the laboratory's ability to meet them. The Vice President is responsible for technical communication and liaison with the clients. He also has responsibility for providing proposal preparation and pricing strategies for potential projects and for technical communication and liaison with the clients.

LABORATORY DIRECTOR - The Laboratory Director is responsible for ensuring that Microseeps' goals of providing accurate and verifiable analyses are met. To do this, it will be the Laboratory Director's responsibility to ensure that all laboratory personnel have the required qualifications and training for their positions. Once qualified personnel are in place, the Laboratory Director, in conjunction with the Quality Assurance Manager, will be responsible for assuring that all employees are thoroughly familiar with the Quality Assurance Manual and accepted laboratory practices.

The Laboratory Director oversees the daily management of the laboratory staff. A major

component of this particular responsibility is the integrity of laboratory reports. The Laboratory Director, or a qualified designee, will review and approve all outgoing reports.

Included in the Laboratory Director's responsibilities is scheduling work and conforming to holding times. The Laboratory Director will schedule work to meet the individual demands of the client and conform to accepted holding time requirements. The Laboratory Director approves analytical results and reports for work performed by the laboratory.

TECHNICAL DIRECTOR - The Technical Director is responsible for ensuring that the data produced by the laboratory are technically sound. He provides assistance to personnel to resolve technical problems within the laboratory groups. He is also responsible for oversight of instrument repairs to ensure that they are provided in a timely and cost effective manner.

QUALITY ASSURANCE COORDINATOR - The QA Coordinator is responsible for assuring that the requirements of the Quality Assurance Manual and the associated Standard Operating Procedures are strictly followed. This is accomplished by: reviewing data validation procedures, alerting the analysts should the need for corrective action exist, performing internal audits, establishing a periodic schedule for analyzing performance evaluation samples, and maintaining QC records. If the need arises for project specific QA/QC plans, it will be the QA Coordinator's responsibility to develop them.

PROJECT MANAGER - the Vice President of Customer Service assigns the position of Project Manager for specific projects. Projects may require a specifically assigned manager because of the unusual nature of the project, complexity of the analytical techniques, special reporting requirements, or the need to coordinate activities in several laboratories.

The Project Manager will alert the Laboratory Director if problems arise in meeting schedules or sample holding times. It is also the Project Managers responsibility to assure that work on the project is performed in accordance with project-specified protocols and following project specific QC requirements

ANALYSTS - Analysts are responsible for performing all analyses as required and following the required Quality Control procedures demanded by the analytical method or technique. To properly perform analyses, the analyst must have a thorough understanding of the Quality Assurance Manual and associated Standard Operating Procedures. They are also responsible for initiating system or method corrective action should they become aware of a malfunction. Initiation of corrective action requires proper notification of the Laboratory Director and Quality Assurance Manager as discussed later in the manual.

SAMPLE CUSTODIAN - The Sample Custodian is responsible for samples from their receipt to their disposal. Included in the responsibility for samples are ensuring that storage and documentation requirements are met. It is incumbent that the Sample Custodian notifies the Laboratory Director should there be any discrepancies or

irregularities with samples when they are received.

3.3 Qualifications

Job assignments are made by the Laboratory Director based upon specific educational and experience requirements. Table 3-1 defines the requirements for various job classifications. Classifications for employees that have an impact on data quality are provided. Some of the positions described previously are classified according to the requirements detailed in Table 3-1, but are given different job titles such as QA Manager.

3.4 Training

Training for laboratory personnel is accomplished at several levels. Areas for which training is conducted include laboratory safety, analytical procedures, quality assurance requirements, and project specific requirements.

3.4.1 In-house Training

3.4.1.1 Orientation

Orientation is conducted to familiarize new employees with company policies, expectations, quality assurance procedures, fellow employees, and laboratory safety. The Laboratory Director is responsible for the orientation.

3.4.1.2 Task Training

Task training must be successfully completed for employees to perform the following tasks without direct supervision:

Sample log-in
Sample preparation and analysis
Data reporting

Task training is conducted by the Laboratory Director, QA Manager, or experienced analyst. During this procedure, the trainer works closely with the trainee to ensure that all pertinent points of procedures and reference materials are addressed. For training to be considered complete, proficiency in the task must be demonstrated. For sample preparation and analysis, proficiency is demonstrated through the use of known reference materials in accordance with the Standard Operating Procedure for Administering and Documenting Training in the Laboratory, SOP-ADM2.

If revisions are made to procedures dealing with sample log-in, sample preparation and analysis, and data reporting, qualified personnel are retrained by reading the revisions and documenting their understanding. Also analysts must also successfully analyze performance evaluation samples to maintain qualification.
The employee's signature on the SOP-ADM2 Read Form and the Analyst Training Record documents the training. The QA Manager maintains training and orientation records.

3.4.1.3 Project Specific Training

Personnel assigned to non-routine or complex projects receive project specific training before the project begins. The assigned project manager conducts the training and documents attendance at these sessions by completing the analyst training record.

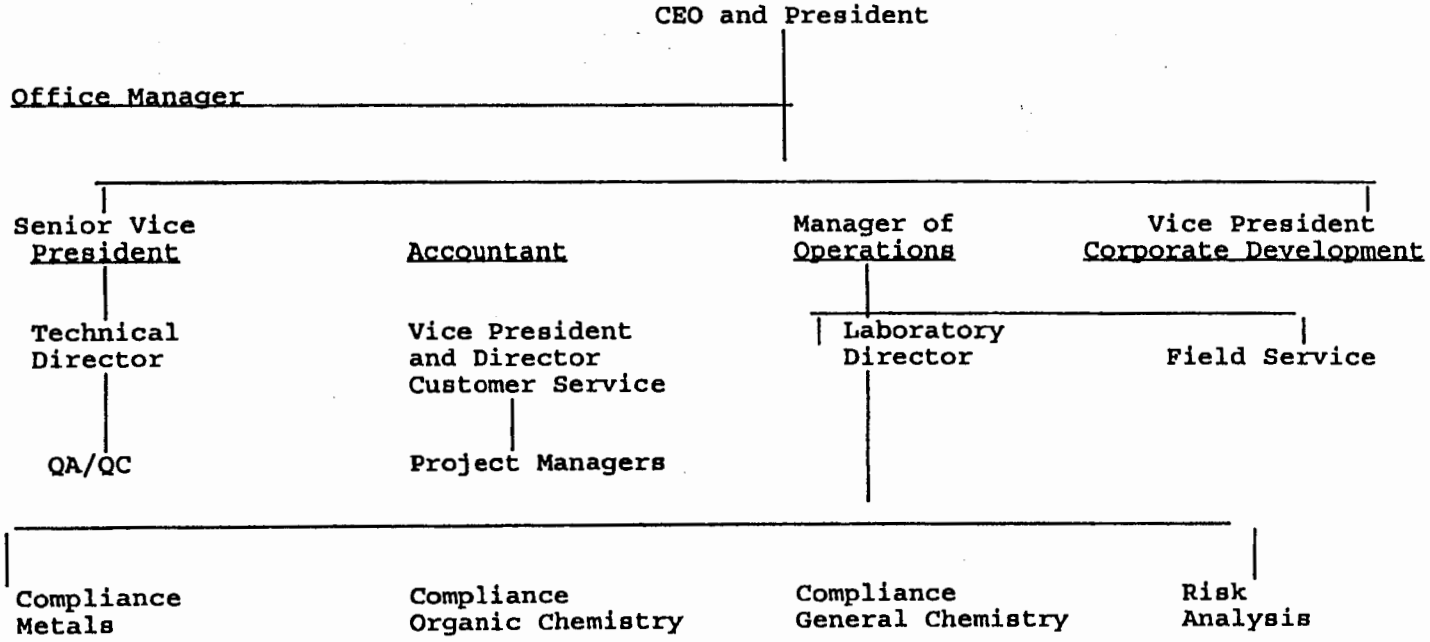
3.4.2 External Training

Employees are encouraged to continue their education through the use of symposia and seminars conducted by professional societies, regulatory agencies, and equipment manufacturers. These courses serve as one way for laboratory personnel to remain current on regulatory trends, analytical procedures, and advances in instrumentation. Documentation of external training will be added to the analysts' training records.

Table 3-1
Personnel Qualifications

TITLE	KNOWLEDGE AND EXPERIENCE
Laboratory Assistant	High school diploma
Laboratory Technician	Technical school certificate or Technical program certificate or 1 year laboratory experience
Analyst	Associate degree or 4 years laboratory experience
Chemist	Chemistry or related sciences degree and 2 years laboratory experience or equivalent related experience
Laboratory Director	Chemistry or related sciences degree And 7 years laboratory experience

Table 3-2
Microseeps, Inc.
Organizational Chart



4.0 SAMPLE MANAGEMENT

Reliable and defensible analytical data begins in the field. Usually Microseeps, Inc. is not responsible for sample collection, however the laboratory is responsible for activities that support the sample collection process. These activities include providing the appropriate sample containers and maintaining sample integrity once samples are received. The purpose of this section is to establish procedures for the type and use of sample containers, the preservation of the samples, and most importantly sample custody.

4.1 Sample Containers and Preservation

In order to provide the best possible service for the client, the laboratory can provide the containers used for sampling, if requested. They are provided in advance of the sampling event and in most cases have preservatives added. The type of container that is used and the type of preservation are dictated by regulation.

4.1.1 Sample Container Construction

Sample containers are constructed of either polyethylene or glass. To ensure sample integrity, containers must be clean and free of contamination. Containers may either be purchased that are pre-cleaned and ready for use, or cleaned by the lab. In addition, certified clean containers can be purchased. Microseeps purchases pre-cleaned VOA vials and oil and grease bottles. All other bottles are purchased uncertified, but kept in a contaminate-free area.

4.1.2 Preservation of Samples

Preservatives are added to many samples during collection to stabilize the sample and maintain its integrity. These preservatives usually are included in the sample container when the analytical lab provides it. The use of preservatives is also dictated by regulation. Tables 4-1 through 4-4 list, by regulation, the type of container and the preservative that is to be used. Preservatives are prepared by an analyst and documented in the standards logbook to facilitate tracking of the preservative.

4.2 Sample Custody

A key component to providing legally defensible data is sample custody. In many cases, it is necessary to trace to sample handling from collection, through delivery to the laboratory and analysis, to final disposal. If Microseeps does not provide sample collection services, sample custody begins when samples are received at the laboratory or picked up at a client site by a Microseeps employee.

4.2.1 Sample Custody and Documentation in the Field

If Microseeps performs the sampling function, a single person from the field team is designated to serve as field sample custodian for onsite operations. It is the sample custodian's responsibility to ensure that the sample labels and chain of custody forms are complete and accurate. Microseeps provides chain of custody forms with every bottle shipment. An example of this form is included as Figure 4-1. These forms, or a client-supplied equivalent, should accompany every sample shipment to the laboratory. This data should also be included in a field notebook. The field custodian is also responsible for properly storing the sample onsite prior to shipment to the laboratory. This may include keeping the samples on ice. The field sample custodian will also prepare the samples for shipment to the laboratory. Care should be taken to ensure that the samples are packaged to prevent breakage during shipment. Ice should be added to the coolers to maintain the samples are 4°C during shipment. Glass containers should not be packed so that they are in direct contact with ice. Direct contact with the ice may cause the samples to freeze and break the containers.

4.2.2 Sample Custody in the Laboratory

Sample custody at the laboratory includes several distinct functions. These functions include sample receipt, sample inspection, documentation reconciliation, resolution of irregularities, sample log-in, information dissemination, custody transfer, sample storage and recovery, and sample disposal. Each of these functions is described in more detail in the following sections.

4.2.2.1 Sample Receipt

Samples are either hand carried to the laboratory or shipped using commercial carriers such as UPS and Federal Express, and the United State Postal Service. The sample custodian or designee signs for each shipment and a copy of the shipping documents is retained.

4.2.2.2 Sample Inspection

Sample shipments are routinely opened and inspected immediately; however, some instances such as off-hour deliveries may prevent this. Prior to opening the shipping container, it is inspected for damage. The log-in clerk or designee notes if custody seals are present and whether they are intact.

Once the shipping container is visually inspected, the container may be opened under a fume hood. Upon opening the container, the chain of custody form is removed and the cooler temperature is checked. The temperature is measured by inserting a thermometer into the cooler, closing the lid and waiting a minimum of five (5) minutes for the reading to stabilize. At the end of the 5 minutes, the temperature is read and recorded on the chain of custody form.

After the cooler temperature is recorded, the individual sample containers are removed and inspected for breakage, cracking, or leaking. If the sample containers are intact, they may be moved to a laboratory bench to continue the log-in process. If there is any evidence of breaking, cracking, noxious fumes, etc., the entire log-in procedure is performed under the hood. Any samples received in less than perfect condition are noted on the chain of custody form.

4.2.2.3 Document Reconciliation

Once all of the samples have been removed from the shipping container, they are quickly counted and compared to the field chain of custody. If the actual number of containers received is less that the total noted on the chain of custody, the shipping container and packing materials are inspected to confirm that a sample container has not been overlooked.

The information recorded on the sample labels is compared to the field chain of custody. Specific items that are noted include the sample description, date and time of collection, and number of containers submitted for analysis. Any discrepancies are noted on the field chain of custody form. The form is then signed and dated. If the shipment is complete and accurate as received, the sample log-in procedure continues. If any problems have been noted with the shipment, the field chain of custody and any additional documents generated to note the discrepancies are forwarded to the Project Manager or Laboratory Director for resolution. Samples are not logged in until all discrepancies are resolved.

4.2.2.4 Resolution of Irregularities

If any problems are noted with a sample shipment, they are noted and brought to the attention of the Project Manager and/or Laboratory Director for resolution.

The client is contacted and the conversation is recorded in a bound notebook or a phone log. The information that should be recorded includes the name of the contact, date and time of the conversation, and the corrective action that should be implemented.

Several possibilities may exist for resolving sample shipment problems. All decisions are the client's responsibility. Once a resolution is determined, the solution is noted and one of the following actions will occur:

The log-in process will continue
The sample(s) will be returned
The sample(s) will be disposed of

Throughout the problem resolution process, the sample will either be kept in a secure area or will be in view of the sample custodian. Any record generated during this process becomes a part of the client's permanent file.

4.2.2.5 Sample Log-in

Because of the critical nature of many analyses, sample log-in represents the central point around which all other laboratory functions revolve. The staff responsible for logging in samples must do so efficiently and accurately so that sample preparations and analysis can be completed as quickly as possible. Sample log-in is initiated once the shipment inspection is completed and any problems are resolved. Samples for a complete job may be received in a single shipment or over a period ranging from several days or months. To facilitate the log-in process, a single job for the laboratory will consist of all samples received from a single client from one site in a single day. The one exception to this rule will involve samples requiring CLP data packages.

Sample and project information from field chain of custody forms as well as any phone logs that may pertain to the project is hand written into a sample receipt logbook. See Figure 4-5. The following information is recorded.

Client name and number
Date received
Date sampled

Date due
Sample description
Matrix
Required analyses
Bottles received

For samples requiring CLP data packages, samples will be separated into jobs based on Sample Delivery Groups (SDGs). An SDG consists of up to twenty field samples received from a single site over a maximum 14-day period.

4.2.2.5.1 Entry in LIMS

Samples will be logged into the LIMS using information contained on the field chain of custody forms as well as any phone logs that may pertain to the project. The detailed procedure for logging samples into the LIMS are defined in the laboratory's Standard Operating Procedures (SOPs).

Samples are logged into the system in the order in which they are received. The following information is required to enter samples into the LIMS:

- Client name and number
- Date received
- Date sampled
- Date due
- Sample description
- Matrix
- Required analyses
- Bottles received

4.2.2.5.2 Sample Labeling

Once all of the data is entered into the LIMS, the system generates an internal chain of custody form (Figure 4-2). These forms are sequentially numbered by the LIMS and are used to track the progress of the project through the laboratory. All documents used to log-in the samples are attached and become part of the permanent records.

Simultaneously with generating the internal chain of custody forms, the LIMS generates bar-coded labels for each sample bottle received. The sample labels contain the client and sample number, sample matrix, bottle type, and the dates that the samples were collected and received at the laboratory.

The labels are placed on the sample bottles and once again checked for accuracy. If they are correct, the samples are transferred to the appropriate storage areas.

4.2.2.6 **Information Disemination**

Work lists for each individual laboratory group are printed each morning. The work lists summarize the client and sample numbers, required analyses, and due dates. The Laboratory Director reviews the information. If correct, the information is forwarded to the responsible laboratory personnel. Any special notes or instructions are added to the printout.

4.2.2.7 **Custody Transfer**

All samples are maintained in access-controlled areas until work is started. The person responsible for either the sample preparation or analysis will retrieve the sample(s) from the storage area and return them when the function is complete.

For samples requiring locked storage, the sample log-in staff initiates a sample tracking record (Figure 4-3). Entries are made to the form each time a sample is removed or returned to the storage areas. Whenever sample preparations are completed, i.e. organic extractions and metals digestions, the sample preparation group adds them to the tracking record.

4.2.2.8 **Sample Storage and Recovery**

Samples are placed into a temperature controlled walk-in cooler that is maintained at 4°C± 2°C. The sample custodian records the cooler temperature each morning and evening. A temperature log is maintained. Figure 4-4.

Extract storage is tracked by the use of a Preparation Log Form, Figure 4-5. This form accompanies the sample extracts to the appropriate analytical group. The extracts are stored in refrigerators located in the analytical

laboratory. The analytical staff maintains the temperature logs for these refrigerators, Figure 4-6.

Sample storage areas are checked on a regular basis to insure that completed samples can be moved from a primary to a secondary storage area. The secondary storage area is not temperature controlled. Samples are stored in secondary storage areas for a minimum of thirty (30) days following the date an analytical report is generated.

4.2.2.9 Sample Disposal

Regulations are continually changing regarding sample disposal requirements. Microseeps monitors these regulations to ensure practices within the laboratory remain compliant. In general, drinking water and wastewater samples are flushed down the drain, provided that permit limits are not exceeded. Other samples are disposed of using commercial waste disposal firms. In some instances, sample remnants are returned to the client.

4.3 Record Retention

All raw data, reports, and invoices for analytical projects are kept for a full seven (7) years. After this time the records are destroyed. Records are kept onsite.

The LIMS database is backed up weekly. The tape on which this backup is stored is maintained off-site.

TABLE 4-1

Container, Preservation and Holding Time Requirements
NPDWR
(Based on EPA-814B-92-002)

Parameter	Volume	Container[1]	Preservative	Maximum Holding Time[2]
Alkalinity	100 ml	P, G	Cool 4°C	14 days
Chloride	100 ml	P, G	None	28 days
Cyanide	500 ml	P, G	NaOH to pH >12, Cool 4°C[3]	14 days
Fluoride	100 ml	P, G	None	28 days
Nitrate (only)	50 ml	P, G	Cool 4°C	28 days
Nitrate/Nitrite, Total	50 ml	P, G	H_2SO_4 to pH <2	28 days
Nitrite	50 ml	P, G	Cool 4°C	48 hours
Orthophosphate	100 ml	P, G	Filter immediately, Cool 4°C	48 hours
pH	100 ml	P, G	None	Analyze immediately[4]
Total Dissolved Solids	100 ml	P, G	Cool 4°C	7 days
Turbidity	100 ml	P, G	Cool 4°C	48 hours
Metals except Mercury[5]	200 ml	P, G	HNO_3 to pH <2	6 months
Mercury	100 ml	P, G	HNO_3 to pH <2	28 days
Volatile Organics	100 ml	G/TLS	Ascorbic acid or Sodium thiosulfate[6] 1:1 HCl to pH <2, Cool 4°C	14 days

TABLE 4-1 NOTES

(1) P - polyethylene G- glass TLS - Teflon-lined septum

(2) Holding time is measured from time of sample collection. Samples should be analyzed as soon as possible after collection

(3) Add 0.6 g ascorbic acid if residual chlorine present.

(4) Within 15 minutes of sample collection.

(5) Arsenic, Barium, Cadmium, Calcium, Chromium, Copper, Lead, Selenium, Silver, Sodium.

(6) 17 mg/40 mL ascorbic acid or 3 mg/40 mL sodium thiosulfate.

TABLE 4-2

Container, Preservation and Holding Time Requirements
Aqueous Samples - NPDES AND RCRA
(Based on 40 CFR Part 136.3, Table II and SW-846 3rd ed. Revision 1, Table 2-21)

Parameter	Volume	Container[1]	Preservative[2][3]	Maximum Holding Time[4]
INORGANICS				
Acidity	100 ml	P, G	Cool, 4°C	14 days
Alkalinity	100 ml	P, G	Cool, 4°C	14 days
BOD	300 ml	P, G	Cool, 4°C	48 hours
Bromide	100 ml	P, G	None	28 days
Chloride	100 ml	P, G	None	28 days
Chromium, Hexavalent	50 ml	P, G	Cool 4°C	24 hours
COD	50 ml	P, G	Cool 4°C;H_2SO_4 to pH <2	28 days
Color	50 ml	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	500 ml	P, G	Cool, 4°C; NaOH to pH >12[5]	14 days
Fluoride	100 ml	P	None	28 days
Hardness	100 ml	P, G	HNO_3 to pH <2	6 months
MBAS (Surfactants)	100 ml	P, G	Cool, 4°C	48 hours
Mercury	100 ml	P, G	HNO_3 to pH <2	NPDES-28 days RCRA-13 days in plastic; 38 days in glass
Metals, except Cr^{+6} and Mercury	200 ml	P, G	HNO_3 to pH <2	6 months

TABLE 4-2
Container, Preservation and Holding Time Requirements
Aqueous Samples - NPDES and RCRA
(Continued)

Parameter	Volume	Container[1]	Preservative[2]	Maximum Holding Time[4]
Nitrogen Ammonia	100 ml	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Kjeldahl, Total	100 ml	P,G	Cool, 4°C; H_2SO4 to pH <2	28 days
Nitrate-Nitrite	50 ml	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Nitrate	50 ml	P, G	Cool, 4°C	48 hours
Nitrite	50 ml	P, G	Cool, 4°C	48 hours
Odor	100 ml	G	None	24 hours
Oil & Grease	1000 ml	AG	Cool, 4°C; H_2SO_4 to pH <2	28 days
Organic Carbon	50 ml	P, G	Cool, 4°C; H_2SO_4 to pH <2	24 days
Oxygen, Dissolved by Probe	200 ml	G bottle and top	None	Analyze immediately
by Winkler Titration		G bottle and top	Fix on site and store in dark	8 hours
pH	100 ml	P, G	None	Analyze immediately
Phenols	500 ml	AG	Cool, 4°C; H_2SO_4 to pH <2	28 days

TABLE 4-2
Container, Preservation and Holding Time Requirements
Aqueous Samples - NPDES and RCRA
(Continued)

Parameter	Volume	Container[1]	Preservative[2][3]	Maximum Holding Time[4]
Phosphorus				
Orthophosphate	100 ml	P, G	Filter immediately; Cool, 4°C	48 hours
Total	100 ml	P, G	Cool, 4°C; H_2SO_4 to pH <2	28 days
Solids				
Dissolved	100 ml	P, G	Cool, 4°C	7 days
Total	100 ml	P, G	Cool, 4°C	7 days
Suspended	100 ml	P, G	Cool, 4°C	7 days
Settleable	100 ml	P, G	Cool, 4°C	48 hours
Volatile	100 ml	P, G	Cool, 4°C	7 days
Specific Conductance	100 ml	P, G	Cool, 4°C	28 days
Sulfate	100 ml	P, G	Cool, 4°C	28 days
Sulfide	100 ml	P, G	Cool, 4°C; Zinc Acetate & NaOH to pH >9	7 days
Sulfite	100 ml	P, G	None	Analyze immediately

TABLE 4-2
Container, Preservation and Holding Time Requirements
Aqueous Samples - NPDES and RCRA
(Continued)

Parameter	Volume	Container[1]	Preservative[2][3]	Maximum Holding Time[4]
TOX	500 ml	AG with TLC	Cool, 4°C, add 1 ml H_2SO_4 pH <2	7 days
Turbidity	100 ml	P, G	Cool, 4°C	48 hours
ORGANIC CHEMISTRY TESTS				
Purgeable Halocarbons	100 ml	G with TLS	Cool, 4°C	14 days
Purgeable Aromatics	100 ml	G with TLS	4 drops conc. HCl/ 40 ml vial; Cool, 4°C[8]	14 days (7 days w/o HCl pres.)
Gasoline Range Organics	100 ml	G with TLS	4 drops conc. HCl/ 40 ml vial; Cool, 4°C	14 days (7 days w/o HCl pres.)
PCBs	1000 ml	G with TLC	Cool, 4°C[7]	7 days until extraction, 40 days after extraction

TABLE 4-2

Container, Preservation and Holding Time Requirements
Aqueous Samples - NPDES and RCRA
(Continued)

Parameter	Volume	Container[1]	Preservative[2][3]	Maximum Holding Time[4]
Pesticides	1000 ml	G with TLC	Cool, 4°C; pH 5-9	pH 5-9: 7 days until extraction; pH <5 or >9: 3 days until extraction; 40 days after extraction
Phenols	1000 ml	G with TLC	Cool, 4°C[7,8]	7 days until extraction, 40 days after extraction

TABLE 4-2

Container, Preservation and Holding Time Requirements
Aqueous Samples - NPDES and RCRA
(Continued)

Parameter	Volume	Container[1]	Preservative[2,3]	Maximum Holding Time[4]
Polynuclear Aromatic Hydrocarbons	1000 ml	G with TLC	Cool, 4°C,[7,8]; store in dark	7 days until extraction, 40 days after extraction
Herbicides	1000 ml	G with TLC	Cool, 4°C, [7]	7 days until extraction, 40 days after extraction
Diesel Range Organics	1000 ml	G with TLC	5 mls, 1:1 HCl; Cool, 4°C	7 days until extraction, 30 days after extraction

TABLE 4-2 NOTES

1. AG - amber glass
   G - glass
   P - polyethylene
   TLC - Teflon-lined cap
   TLS - Teflon-lined septum

2. Sample preservation should be performed immediately upon sample collection. For composite samples, samples may be preserved by maintaining at 4°C until sample splitting and compositing is completed.

3. If the dissolved content is to be measured, samples should be filtered on site immediately before adding preservatives.

4. The holding times listed are the maximum times that samples may be held before analysis and still be considered valid under EPA regulations. Holding times are measured from sampling.

5. Add 0.6 g ascorbic acid/liter sample if residual chlorine is present.

7. pH should be 6-9.

8. Add $Na_2S_2O_3$ if residual chlorine is present (0.008 percent).

TABLE 4-3

Container, Preservation and Holding Time Requirements
Non-Aqueous Samples - NPDES and RCRA
(Based on SW-846 3rd ed., Revision 1, Table 4-1)

Parameter	Volume	Container[1]	Preservative[2][3]	Maximum Holding Time[3]
Matrix				
VOLATILE ORGANICS				
Concentrated Waste	10g	Wide-mouth glass with TLC	None	14 days
Soil/Sediment/Sludge	10 g	Wide-mouth glass with TLC	Cool, 4°C	14 days
SEMIVOLATILE ORGANICS				
Concentrated Waste	10g	Wide-mouth glass with TLC	None	14 days until extraction, 40 days thereafter
Soil/Sediment/Sludge	30g	Wide-mouth glass with TLC	Cool, 4°C	14 days until extraction 40 days thereafter

TABLE 4-3 NOTES

1. TLC = Teflon-lined cap.

2. Soil/sediment and sludge samples should be cooled to 4°C for all parameters.

3. The holding times listed are the maximum times that samples can be held before analysis and
still be considered valid under EPA regulations. Holding time is measured from sampling.

TABLE 4-4

Container, Preservation and Holding Time Requirements
CLP

Parameter	Volume	Container[1]	Preservative[2][3]	Maximum Holding Time[4]
Metals except Mercury	1000 ml	Aqueous: P with PLC	HNO_3 to pH <2	6 months
	4-32 oz.	Non-Aqueous: G with PLC	Cool, 4°C	6 months
Mercury	1000 ml	Aqueous: P with PLC	HNO_3 to pH <2	26 days
	4-32 oz.	Non-Aqueous: G with PLC	Cool, 4°C	26 days
Cyanide	1000 ml	Aqueous: P with PLC	NaOH to pH <12; Cool, 4°C[5]	12 days
	4-32 oz.	Non-Aqueous: G with TLC	Cool, 4°C	12 days
Volatile Organics	2-40 ml vials	Aqueous: G with TLS	Cool, 4°C; Dark	10 days
	4 oz.	Non-Aqueous: G with TLC	Cool, 4°C; Dark	10 days
Semivolatile Organics, Pesticides/PCBs	1/2 gal.	Aqueous: AG with TLC	Cool, 4°C; Dark	5 days until extraction[6], 40 days thereafter
	8-16 oz.	Non-Aqueous: AG with TLC	Cool, 4°C; Dark	10 days until extraction[6]; 40 days thereafter

Table 4-4 NOTES
Container, Preservation and Holding Time Requirements
CLP
(Continued)

1. AG = Amber glass
   G = Glass
   P = Polyethylene
   PLC = Polyethylene-lined cap
   TLC = Teflon-lined cap
   TLS = Teflon-lined septum

2. Sample preservation should be performed immediately upon sample collection. For composite samples, samples may be preserved by maintaining at 4°C until compositing and sample splitting are complete.

3. If the dissolved content is to be measured, samples should be filtered on-site immediately before adding preservatives.

4. The holding times listed are the maximum times that samples may be held before analysis and still be considered valid under EPA regulations. Holding times are measured from the verified time of sample receipt (TVSR) at the laboratory. (Holding time in the field must be minimized when organics and/or cyanide are parameters of interest.)

5. If residual chlorine is present, add 0.6 g ascorbic acid.

6. Separatory and sonication extraction procedures must be completed within the holding time. Continuous extraction procedures (applicable to aqueous samples only) must be started within the holding time.

Figure 4-1
Chain of Custody Form

Phone: (412) 826-5245 Microseeps, Inc. - 220 William Pitt Way - Pittsburgh, PA 15238 Fax No. : (412) 826-3433

Company :
Co. Address :
Proj. Manager:
Proj. Location:
Proj. Number:
Phone # : Fax # :

Sampler's signature :

Parameters Requested

Remks to :
Invoice to :

Cooler ID	Cooler Temp.

Sample ID	Sample Description	Date	Time	Comp.	Grab	# Cont.	Remarks

Relinquished by :	Company :	Date :	Time :	Received by :	Company :	Date :	Time :
Relinquished by :	Company :	Date :	Time :	Received by :	Company :	Date :	Time :
Relinquished by :	Company :	Date :	Time :	Received by :	Company :	Date :	Time :

WHITE COPY : Accompany Samples YELLOW COPY : Laboratory File PINK COPY : Submitter

Figure 4-2
Internal Chain of Custody Form

INTRA-LABORATORY
CHAIN-OF-CUSTODY

Client: ABC Company
1000 Jones Street
Suite 2000
Pittsburgh, PA 15222-

Date: 03/06/95

C-of-C ID No.: 133850

Group Identification: Site D
Client No.: 9900-0 P.O. No.: 030695

Date Sampled: 03/06/95
Date Received: 03/06/95

Sampled By: John Smith

ID or Permit No.: _____

	Field Parameters					Sample Bottles/Custody Seals									
Sample ID	Time Sampled	pH	Cond. umhos/cm	Temp °C	Depth	G. Chem	Metal	O&G	CN	Phenol	N. Caps.	Organ. VOAs	TOX	Biol/ Other	MLI Sample Number
MW-1	11:00	7.0	1000	20	173.55	1 B	2 B	1 B		1 B	1 B			1 B	4
MW-2	12:00	6.8	1500	17	69.98	1 B	2 B	1 B		1 B	1 B			1 B	5
			MATRIX:		Water	EXTRACTION:		Total							

NOTES: Circle (B) for Broken Custody Seals. If NO Seals Draw Single Line thru B. Number indicates Number of Bottles.

ANALYSES REQUESTED:
pH Cond TDS Alk HCO3 Cl NH3 NO3 TPH Phnl Cn Cu-d Mn Mn-d Pb Pb-d Zn Zn-d

NOTES: _____

Transmitted to MLI (Lab) by: _____

Title: _____ Date/Time: _____

Received By: _____ Date/Time: _____

Relinquished By: _____ Date/Time: _____

Received By: _____ Date/Time: _____

Relinquished By: _____ Date/Time: _____

Received By: _____ Date/Time: _____

Relinquished By: _____ Date/Time: _____

Received By: _____ Date/Time: _____

Relinquished By: _____ Date/Time: _____

COMPLETED ANALYSES: [ ] G. Chem. [ ] Metals [ ] Organics
Approx. Price: $[illegible].00 Due Date: 03/18/95

Final Disposition of Samples: DESTROYED RET TO CLIENT DRUM A DRUM B OTHER _____ DATE: _____

Figure 4-3
Sample Tracking Record

Page ___ of ___

Sample Storage Location: _____

Case No./Project Name: _____

SDG Number: _____

Samples Received By: _____ Date/Time

Samples Transferred To: _____ Date/Time

Sample Numbers	Sample Containers
_____	_____
_____	_____
_____	_____
_____	_____
_____	_____

Container/Fraction	MLI Sample Numbers	Removed from Storage			Returned to Storage		
		By	Date	Time	By	Date	Time

5.0 FACILITIES AND INSTRUMENTATION

5.1 Facility Description

Microseeps is located in the University of Pittsburgh's Applied Research Center (UPARC). This complex formerly housed Gulf Oil's research facility. The various laboratory work areas are segregated as depicted in Figure 5-1 and 5-2. This segregation helps to prevent cross-contamination between the preparation and analytical laboratories.

Access into the complex is controlled through a central guard gate. All visitors must sign in at the gate before they can proceed to the physical building, which houses the laboratory areas. Once inside the Microseeps building, visitors are received at a central office location. The visitor is then escorted to the Microseeps employee or laboratory they intend to visit.

The laboratory areas are controlled through keyed entry to prevent employees from other firms housed in the complex from gaining access to Microseeps's laboratories. Each employee is issued a key that will open any door to rooms occupied by Microseeps. During normal working hours, the laboratory areas are kept unlocked; however, during the evening and over the weekends, the rooms are locked to prevent admittance by unauthorized personnel. Random checks are made to ensure that facility keys are in the possession of the designated personnel.

5.2 Instrumentation

Instrumentation must be properly calibrated and maintained to produce reliable and reproducible results. This section of the QA manual defines minimally acceptable standards for installation, calibration, and maintenance of analytical instruments used in the laboratory. Fully detailed procedures are instrument specific and are available in the appropriate SOP's.

5.2.1 Installation and Set-up

All new instrumentation must be included in the QA program prior to use for sample analysis. When new equipment is ordered the Laboratory and Technical Directors should design a plan that defines the preparations that need to be made at the laboratory to accommodate the equipment. This plan should include descriptions of facility modifications that may be required, person responsible for installation (manufacturer or Microseeps employee), performance criteria that needs to be met, and training procedures that will be followed.

Data generated during installation and set-up will be included in the maintenance log for the instrument. This data may become important later for troubleshooting and diagnostics checks. Operational manuals supplied by the manufacturer will be maintained in the laboratory for reference.

5.2.2 Calibration

Calibration procedures for instrumentation must be thoroughly documented and routinely followed to provide some assurance that the data produced are reliable and accurate. There are two types of calibrations performed in the laboratory, initial and continuing.

5.2.2.1 Initial Calibration

Initial calibration involves comparing instrumental response to various concentration levels of the analytes of interest. The calibration curve will contain a minimum of three points, excluding a blank. The lowest point of the curve should be approximately 2 times the instrument detection limit. The most concentrated standard should be at or near the upper limit of the linear range. All standards should be prepared from reagents traceable to NIST. For organic analyses, surrogates are added to each blank and standard. In addition, internal standards are also added for some organic analyses.

The analyst records the concentration of the working solutions, date of analysis, and instrument responses in the raw data notebook and/or run logs. When analysis of the calibration curve is completed, the correlation coefficient is calculated. The correlation coefficient must be greater than or equal to 0.995 in order for the curve to be valid. If the criteria are not met, one of more points of calibration curve may be reanalyzed. If the curve still fails to meet the criteria, the analyst should plot the curve. If the curve is not significantly bowed or "S" shaped and it passes near the origin, the samples may be quantitated against the curve.

Gas chromatographs/mass spectrometers must successfully pass a tune for each 12-hour period in which the instrument is in use. The criteria that must be met are detailed in Table 5-1.

Response factors will be calculated for analyses using gas chromatography. For analyses using external standardization, the factor will be calculated by dividing the concentration by the peak area. Whenever, internal standardization is used, the response factor is calculated using the following formula.

$RF = (A_s C_{is})/(A_{is} C_s)$ where:

A_s = Area for the analyte of interest

A_{is} = Area for the internal standard
C_s = Concentration for the standard
C_{is} = Internal standard concentration

For multi-response pesticides and PCBs, the total area for all peaks will be used for quantitation.

The percent relative standard deviation (%RSD) for the response factors must be less than or equal to 20 percent for the factors to be considered valid.

All calibration curves will be confirmed for accuracy by analyzing a calibration verification (ICV) standard. This standard will contain all of the analytes of interest at a known concentration, but is prepared from a different source that the calibration standards. The recovery of this standard should be within 10% of its true value. The ICV is followed by an initial calibration blank (ICB) to ensure that system contaminants or carry over are not present.

5.2.2.2 Continuing Calibration

To verify the stability of the calibration curve during the analytical run, continuing calibration (CCV) checks are analyzed at regular intervals. These standards consist of a midrange standard containing all of the target analytes as well as internal standards and surrogates, as applicable. The standards are analyzed after every 10 determinations and at the end of the run. Recovery for the standard must be within ten percent of the true value for inorganics and fifteen percent for organics to be acceptable. If the acceptance criteria are not met, the run is stopped, the system re-calibrated, and all samples analyzed since the last acceptable CCV are reanalyzed.

5.2.2.3 Calibration Frequency and Documentation

5.2.2.3.1 pH meters

The pH meter and electrode assembly are calibrated using NIST certified buffers of 7.0 and either 4.00 or 10.00 which are commercially available. The assembly is calibrated at the beginning and end of each series of samples. If more than 20 samples are analyzed at one time, a one-point calibration check at pH 7 is conducted after every 20 samples. Records are maintained on the analytical data sheet.

5.2.2.3.2 Balances

Daily calibration checks are made by the analysts in range of use. Calibration checks from 0 to 100 grams are performed to verify the

accuracy of the balances. Weekly records are maintained in logbooks which indicate the date, actual value, observed value, the balance identification, acceptability, and the technicians initials.

5.2.2.3.3 Selective Ion Meters and Electrodes
Standards are run prior to the actual sample determinations. Records are maintained on the analytical data sheet.

A calibration set of three standards, formulated in de-ionized water from reagent grade chemicals, and a blank are run before each sample set analyzed by a specific ion meter or specific ion electrode. The standard curve is prepared by plotting the concentration readings obtained versus the true concentration. A check standard is run after every 20 samples. The check standard must be within 25 percent of the true value.

5.2.2.3.4 Inductively Coupled Plasma (ICP) and Atomic Absorption
ICP standards are purchased from a commercial vendor. The instrumental design of the ICP provides for a minimum of a two-point calibration. Atomic Absorption Calibration utilizes a three to five point curve depending upon which element is being determined. Initial calibrations are performed prior to the actual analysis of samples. A calibration verification check sample is analyzed after every 10 samples. The recovery of the ICV standard should be within 10% of its true value. for both ICP and Atomic Absorption analyses. Records are maintained on the printout obtained from the instruments.

5.2.2.4 Corrective Action for Calibration Failure
In the event that an instrument cannot be calibrated the Laboratory Director or Technical Director may decide to take the instrument out of service or to repair the instrument using in-house personnel. If the instrument is taken out of service an out of service sign is placed on it.

5.2.3 Instrument Maintenance

Maintenance of analytical instruments includes both routinely scheduled preventive maintenance as well as repair due to malfunction failure. Instrument maintenance follows specifications defined by the manufacturer and criteria specified by approved analytical procedures. Maintenance activities for instrumentation is documented in both run and maintenance logs. This documentation becomes a part of the laboratory's permanent records..

5.2.3.1 Preventive Maintenance

Routine preventive maintenance is performed at regularly scheduled intervals to

reduce the potential for instrument malfunction. The schedule for preventive maintenance is based upon the manufacturer's recommendations. If more frequent maintenance is required, the schedule will be amended. A more detailed description of the types of maintenance and frequency of each schedule is outlined in Figures 5-3A through 5-3I.

The Laboratory Director is responsible for providing the necessary time for preventive maintenance. Thorough and complete documentation is required whether the maintenance is performed by laboratory personnel or outside vendors. The Laboratory Director and/or QA Manager is responsible for ensuring that the records are accurate and comply with Standards Operating Procedures. Prior to sample analysis, instrument verification and re-calibration must take place.

5.2.3.2 Instrument Repair

Unexpected repairs resulting from instrument failure are scheduled immediately after the malfunction is observed. Instrument failures are detected through direct observations and by evaluation of the response of verification standards throughout the analytical run. The Laboratory Director is responsible for deciding if laboratory personnel can make the repair or if an outside contractor is required.

Data obtained during instrument failure are not entered into the LIMS for reporting to the client. Complete records of the repairs are maintained in the instrument maintenance logs. These records may include notes taken by laboratory personnel during repair and a copy of the service call record.

Acceptable instrument performance must be verified before samples can be analyzed.

Table 5-1
Tune Criteria for GC/MS Analyses

Volatile Analyses

Mass	Ion Abundance Criteria
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	base peak, 100% relative abundance
96	5 to 9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5 to 9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5 to 9% of mass 176

Semivolatile Analyses

Mass	Ion Abundance Criteria
51	30 to 60% of mass 198
68	less than 2% of mass 69
70	less than 2% of mass 69
127	40 to 60% of mass 198
197	less than 1% of mass 198
198	base peak, 100% relative abundance
199	5 to 9% of mass 198
275	10 to 30% of mass 198
365	greater than 1% of mass 198
441	present but less than mass 443
442	greater than 40% of mass 198
443	17 to 23% of mass 442

Fig
Laboratory Floor Plan

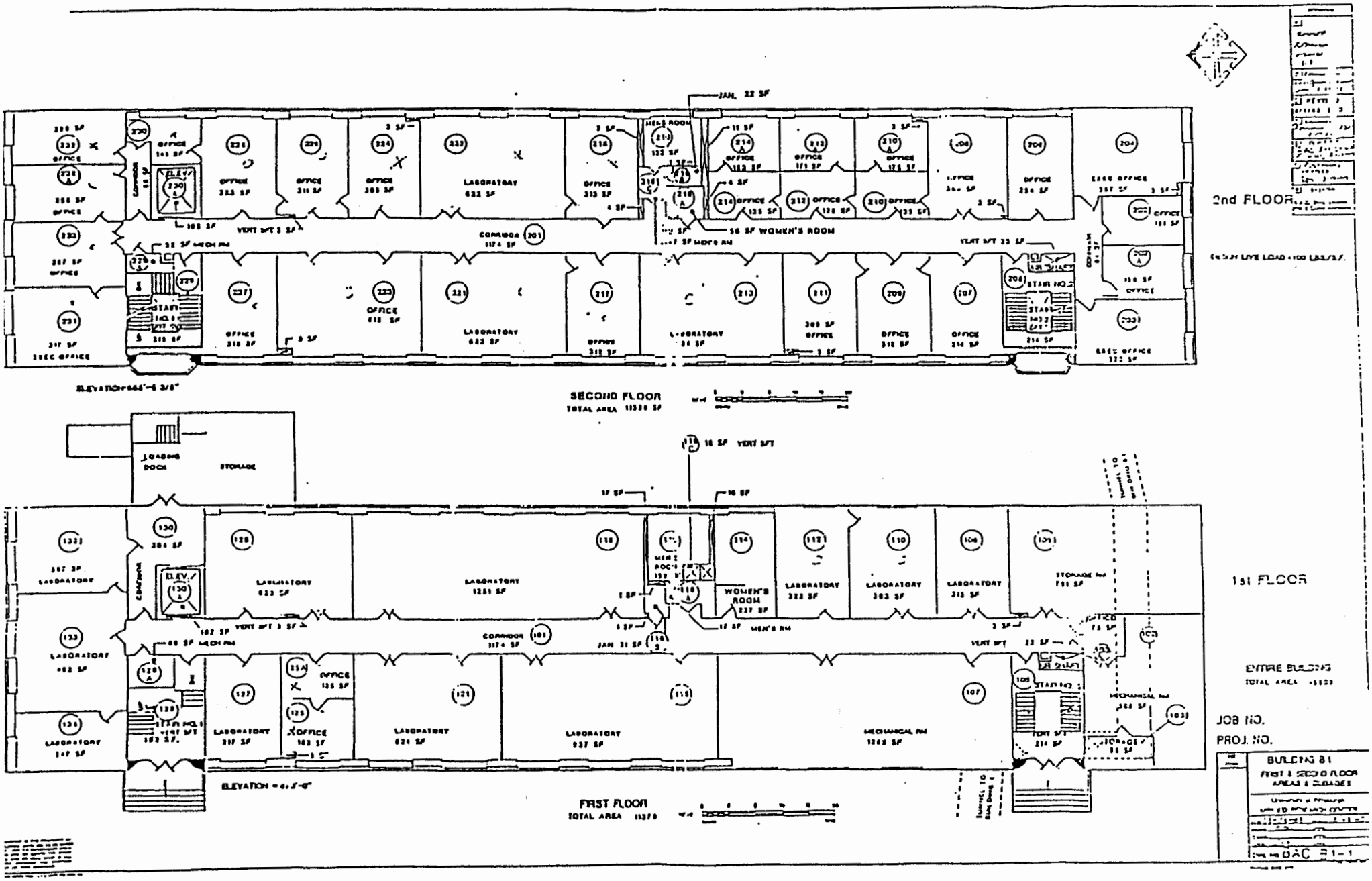


Figure 5-2
Laboratory Floor Plan

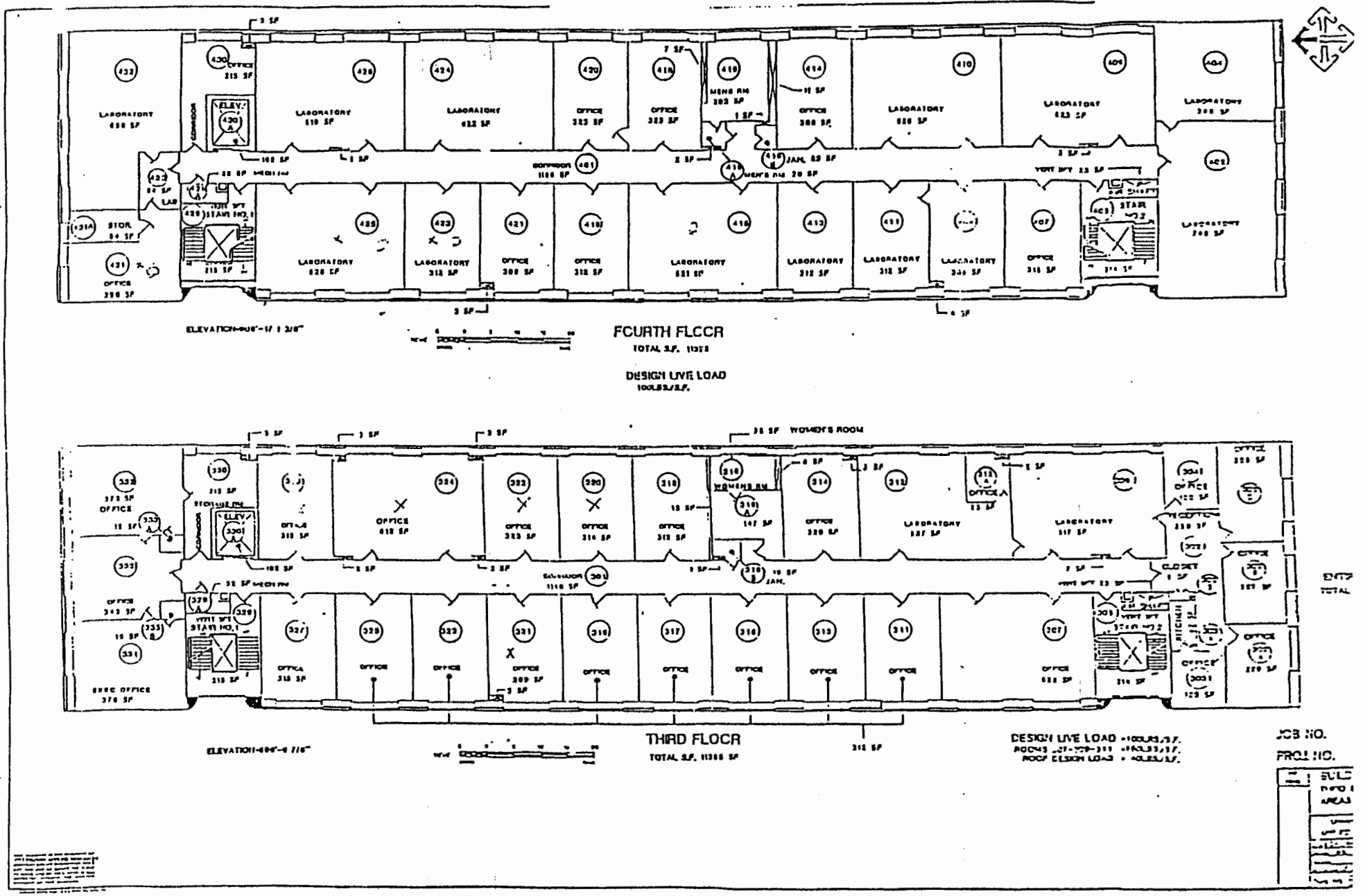


Figure 5-3 A
Microseeps,Inc.
Preventive Maintenance Program
Gas Chromatography/Mass Spectrometry
Volatiles

Instrument ID ___ Model # ___	Frequency
General:	
Check septa, cylinder gas pressure	D
Bake out injector body	2
Check electronics (Voltages, waveforms, etc.)	(3,4)
Check GC temperature calibrations (Injector, oven, detector)	Q
Columns	
Change glass sleeve inserts, shorten ends of columns, check for leaks or replace	W
Mass Spectrometer	
Replace vacuum pump oil and change desiccant	A
Check ion source and analyzer (Dismantle and clean, replace parts as needed)	1
Check mechanical (vacuum pumps, relays, and Gas pressure and flows)	A
Check mass calibration w/FC-43 (perfluorotributylamine)	D
Purge and Trap	
Clean sparger	W
Change Trap	1
Bake Trap	D
Check purge flow	M
Check for leaks	M

Key:

1. Replace as necessary	D	* Daily
2. High background	W	Weekly
3. Loss of sensitivity or failing resolution	M	Monthly
	Q	Quarterly
4. Erratic response	SA	Semi-annually
5. QC failure	A	Annually
6. Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3 B
Microseeps, Inc.
Preventive Maintenance Program
Gas Chromatography/Mass Spectrometry
Semivolatiles

Instrument ID ___ Model # ___	Frequency
Mass Spectrometer	
Replace vacuum pump oil and change desiccant	A
Check ion source and analyzer (Dismantle and clean, replace parts as needed)	1
Check mechanical (vacuum pumps, relays, and Gas pressure flows)	A
Check mass calibration w/FC-43 (perfluorotributylamine)	D
General:	
Check septa, cylinder gas pressure	D
Bake out injector body	2
Check electronics (Voltages, waveforms, etc.)	(3,4)
Check GC temperature calibrations (Injector, oven, detector)	Q
Columns	
Change glass sleeve inserts, shorten ends of columns, check for leaks or replace	W
Purge and Trap	
Clean sparger	W
Change Trap	1
Bake Trap	D
Check purge flow	M
Check for leaks	M

Key:

1.	Replace as necessary	D	*Daily
2.	High background	W	Weekly
3.	Loss of sensitivity or failing resolution	M	Monthly
		Q	Quarterly
4.	Erratic response	SA	Semi-annually
5.	QC failure	A	Annually
6.	Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3C
Microseeps, Inc.
Preventive Maintenance Program
Gas Chromatography

Instrument ID___ Model #___	Frequency
General:	
Check septa, cylinder gas pressure	D
Bake out injector body	2
Check electronics (voltages, waveforms, etc.)	(3,4)
Check GC temperature calibrations (injector, oven, detector)	Q
Columns	
Change glass sleeve inserts, shorten ends of columns, change glass wool plugs check for leaks or replace	W
Electron Capture Detector	
Wipe tests	SA
Hydrogen cleaning	2,3
Returned to factory for cleaning	3,4
Flame Ionization Detector	
Clean	1,2
Replace Flame Tip	1

Key:

1. Replace as necessary	D	*Daily
2. High background	W	Weekly
3. Loss of sensitivity or failing resolution	M	Monthly
	Q	Quarterly
4. Erratic response	SA	Semi-annually
5. QC failure	A	Annually
6. Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3D
Microseeps, Inc.
Preventive Maintenance Program
Metals

Instrument ID	Model #	Frequency
AA Spectrophotometer (Flame)		
Clean nebulizer		SA
Clean quartz windows		W
Burner head cleaned; check tubing, pump and lamps		D (1)
O rings checked		M (1)
Fine Tune; Wavelength;check optics		A
Check electronics		A (3,4)
Instrument ID	**Model #**	**Frequency**
AA Spectrophotometer (Furnace)		
Check graphite tubes		D (1)
Flush autosampler tubing		D
Replace graphite electrodes and shrouds		SA
Clean furnace housing and injector tips		D
Check electronics		A (3,4)
Instrument ID	**Model #**	**Frequency**
AA Spectrophotometer (Cold Vapor)		
Flush tubing (automated systems)		D
Check absorption cell for vitrification		D (1)
Replace or clean quartz cell		3
Check electronics		A (3,4)
Instrument ID	**Model #**	**Frequency**
ICP		
Clean and realign torch		M
Clean nebulizer and spray chamber; Checkperistaltic pump tubing and vacuum pump oil		W (1)
Check entire optical system (mirrors, windows, etc.)		A (3,4)
Check water lines, torch, and gases		D
Check electronics(voltages, waveforms)		SA
Check wavelength calibration		SA
Run interference (interelement) std.		SA

Key:

1. Replace as necessary	D	*Daily
2. High background	W	Weekly
3. Loss of sensitivity or failing resolution	M	Monthly
	Q	Quarterly
4. Erratic response	SA	Semi-annually
5. QC failure	A	Annually
6. Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3E
Microseeps, Inc. Preventive Maintenance Program
General Chemistry

Instrument ID	Model #	Frequency
Infrared Spectrophotometer		
Clean instrument housing		M
Change desiccant and clean cells		Q
Clean windows		M
Instrument ID	**Model #**	**Frequency**
Autoanalyzers		
Check for leaks, flush out system and clean up spills after use		D
Clean sample probe and check all tubing for wear and discoloration		M (1)
Clean optics		Q
Oil sample motor, lubricate gears, clean flow cell		SA
Clean pump rollers, platens and colorimeter filter		M
Instrument ID	**Model #**	**Frequency**
TOX Analyzer		
Clean titration cell; clean inlet and exit tube		W
Clean pyrolysis tube, recoat electrodes		5
Instrument ID	**Model #**	**Frequency**
TOC Analyzer		
Change injection needle, clean injection port, and change catalyst		M
Inspect combustion tube		SA
Instrument ID	**Model #**	**Frequency**
UV/Vis Spectrophotomer		
Check lamp alignment		3,4
Windows cleaned		M
Check and adjust photomultiplier sensitivity and wavelength resolution		A
Replace lamp		4
Clean sample compartment before and after use		D
Check electronics		A (3,4)
Adjust baseline for smoothness through entire wavelength range		SA
Clean cuvettes after each use		D (6)

Figure 5-3E
Microseeps, Inc.
Preventive Maintenance Program
General Chemistry, Continued

Instrument ID	Model #	Frequency
pH and Ion Selective Electrodes		
Probe: Check for cracks, filling solution; check reference junction; clean electrode		D (1)
Check response time		D
Meter: Check batteries and electronics for loose connections and cracked leads		D (1)

Instrument ID	Model #	Frequency
Turbidimeter		
Clean instrument housing		M
Clean cells		D (6)
Instrument ID	Model #	Frequency
Conductivity Meter		
Check batteries and probe cables		D
Replatinize Probe		5
Instrument ID	Model #	Frequency
Dissolved Oxygen Meters		
Probe: Check membrane; filling solution		D (1)
Meter: Check battery level and electronics		D (1)
Instrument ID	Model #	Frequency
Temperature probe		
Check connections, cables		D
Check against calibrated thermometer		D
Instrument ID	Model #	Frequency
Autosamplers		
Check needles and tubing		D (1)
Clean		Q
Instrument ID	Model #	Frequency
Autoclaves		
Gaskets checked		W (1)
Check timing mechanism		SA
Clean interior		M
Sterilization indicator tape		D

Key:

1. Replace as necessary	D	*Daily
2. High background	W	Weekly
3. Loss of sensitivity or failing resolution	M	Monthly
	Q	Quarterly
4. Erratic response	SA	Semi-annually
5. QC failure	A	Annually
6. Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3F
Microseeps, Inc.
Preventive Maintenance Program
Quality Control

Instrument/Activity	Frequency
Refrigerators, Incubators, Ovens	
Clean interior	M
Check temperature	D
Balances	
Clean pan and compartment	D
Check calibration with class S weights	D
Manufacturer or Service Contract (cleaning and calibration)	A
Weights	
Certified calibration of Class S weights	A
Thermometers	
Certified Calibration of NIST thermometer	A
Calibrate working thermometers at the temperature of use against an NIST certified thermometer	A
Logbooks	
Review departmental maintenance, standards, and calibration logbooks	Q

Key:

1. Replace as necessary	D	*Daily
2. High background	W	Weekly
3. Loss of sensitivity or failing resolution	M	Monthly
	Q	Quarterly
4. Erratic response	A	Semi-annually
5. QC failure	A	Annually
6. Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3G
Microseeps, Inc.
Preventive Maintenance Program
Gas Chromatography
Dissolved Gasses

Instrument ID ___ Model # ___	Frequency
General:	
Check septa	D
Check cylinder gas pressure	D
Change Dry Rite on (Carrier gas)	1
Change Activated Carbon (Hydrogen unit)	SA
Bake out Columns at 215°C	D
Check electronics (voltages, waveforms, etc.)	(3,4)
Check GC temperature calibrations (injector, oven, detector)	Q
Columns	
Change glass sleeve inserts, shorten ends of columns, change glass wool plugs check for leaks or replace	W

Key:

1. Replace as necessary	D	*Daily
2. High background	W	Weekly
3. Loss of sensitivity or failing resolution	M	Monthly
	Q	Quarterly
4. Erratic response	SA	Semi-annually
5. QC failure	A	Annually
6. Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3H
Preventive Maintenance Program
Risk Analysis Gas Chromatography
Screening Analysis

Instrument ID _____ Model # _____	Frequency
General:	
Change/Check cylinder gas pressure	D,1
Change Dry Rite on(Air, He, N_2	1
Check detector signal stability	D
Columns (Replace,slow-leak, leak-check, shorten ends, stc.)	3,4,1
Check GC Temperatures (oven, auto sampler, injector, transfer line, etc.)	3,4
Clean autosampler positioning sensors	SA,4
"Wipe-test" ECD detector	A
Clean FID detector	A,3,4

Key:

1.	Replace as necessary	D	Daily
2.	High background	W	Weekly
3.	Loss of sensitivity or failing resolution	M	Monthly
		Q	Quarterly
4.	Erratic response	SA	Semi-annually
5.	QC failure	A	Annually
6.	Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

Figure 5-3 I
Preventive Maintenance Program
Risk Analysis Gas Chromatography
Dissolved Gasses

Instrument ID _____	Model # _____	Frequency
General:		
Check injection septa		D
Change/Check cylinder gas pressure		D
Change Dry Rite on (Air, N_2, He cylinders)		1
Change Activated Carbon (RGD dector)		SA
Bake out Columns at 215°C		D
Check electronics (RGD lamp, voltages, waveforms, etc.)		3,4
Check GC temperature (oven)		3,4
Replace columns		3,4,1
Replace selenoid actuators		3,4,1

Key:

1.	Replace as necessary	D	Daily
2.	High background	W	Weekly
3.	Loss of sensitivity or failing resolution	M	Monthly
		Q	Quarterly
4.	Erratic response	SA	Semi-annually
5.	QC failure	A	Annually
6.	Prior to sampling event		

*Daily is defined as prior to use or a 12-hour period if equipment is run continuously.

6.0 MATERIAL CONTROL

The purpose of this section is to define requirements for the procurement of materials needed to support laboratory operations. The Laboratory Director must review and approve all orders before they are placed.

6.1 Placing Orders

The analyst specifies the materials that need to be ordered on a Purchase Order Form (Figure 6-1). The analyst completes the form by filling in a suggested vendor's name and catalogue number, a description of the item, the quantity requested, the date of the request, and their name. The form is forwarded to the appropriate Manager or Department Director for approval. The Managers and Directors have the discretion to delay the order, obtain equivalent materials from another supplier, or adjust the quantity if it is in the best interest of the laboratory to do so. It is their responsibility to choose an acceptable vendor.

Once the order is approved, the Administrative Assistant places the order.

6.2 Receipt of Materials

Whenever supplies or materials are received, the shipping clerk, Administrative Assistant, or person who placed the order unpacks the shipment and compares the materials received to the packing list. Any discrepancies are noted on the packing list. A call is placed to the vendor to resolve any problems.

Once the order is reconciled with the purchase order, the packing list(s) is attached to the purchase order and is held by the Administrative Assistant until an invoice is received. When the invoice is received, the Administrative Assistant compares it to the packing list and purchase order. If any discrepancies are noted, the vendor is contacted to resolve the problem.

When the invoice, packing list, and purchase order are reconciled, they are forwarded to Accounts Payable for payment. Once payment has been made the documentation is filed in the vendor files.

PURCHASE ORDER
MICROSEEPS

UPARC, 222 William Pitt Way, Pittsburgh, PA 15238
Phone: (412) 826-5245 Fax: (412) 826-3433

chase Order Number: _____
ount Number : _____
er Reference Number: _____

Vending Company: _____
Phone Number: _____
Approval _____

n	Qty	Units	Catalog Number.	Item Description	Size	Manuf.	Unit $	Total $	Accounting Category	Initials
3										
9										
0										
1										
2										
3										
4										
5										

ate Ordered: _____
ate Requested: _____
ate Received: _____

Ordered By: _____
Shipping Requested: _____
Received By: _____

omments: _____

7.0 ANALYTICAL PROCEDURES AND PLANS

The purpose of this section is to define the requirements for developing, modifying, and distributing laboratory procedures and plans. Procedures are designed to provide step-by-step instructions for various laboratory activities. Procedures may be either administrative or analytical in nature. Plans provide the guidance necessary to ensure that a project or activity is completed as established.

7.1 Procedures

7.1.1 Administrative Procedures

Microseeps will prepare and maintain written procedures for all administrative functions that support the laboratory operation. These functions include, but are not limited to, sample bottle preparation, sample log-in and storage, and laboratory security and safety.

The Laboratory Director will be responsible for ensuring that the necessary procedures are prepared. The preparation may be delegated to employees in the work areas; however, ultimate responsibility rests with the Laboratory Director.

Administrative procedures will begin with Section SOP-S1 and SOP-ADM1 of the SOP Manual and will be prepared using the following format:

Title Page
1.0 Purpose and Applicability
2.0 Definitions
3.0 Responsibilities
4.0 Procedure
5.0 Safety
6.0 Equipment and Reagents
7.0 References

Not all sections may be applicable to every procedure.

Administrative procedures are reviewed and approved by the Laboratory Director. The procedures are distributed to each work group, at a minimum.

Revisions are prepared as necessary. All procedures will be reviewed at least every 3 years to ensure that they remain current. Methods are recalled by the Laboratory Director when they are no longer relevant.

7.1.2 Analytical Procedures

Written procedures will be maintained for all laboratory tests designated in the price list and for all major instrumentation. Microseeps will use EPA-approved procedures, when available. If an EPA-approved procedure is unavailable, an industry recognized method will be selected for use or an in house method that includes appropriate QA/QC measures, will be developed and approved by the Laboratory Director and the Technical Director when requested. Client developed methods may be used for analysis, if the proper QA/QC standards are met and if they meet the approval of the Laboratory Director and the Technical Director.

The Laboratory Director will be responsible for preparing all analytical procedures. Analytical procedures will be formatted in the following fashion:

Title Page

1. Purpose and Application
2. Method Summary
3. Apparatus, Materials and Operating Condition
   - 3.1 Apparatus
   - 3.2 Materials
   - 3.3 Operating Conditions
4. Reagents
5. Procedure
   - 5.1 Sample Preparation
   - 5.2 Calibration
   - 5.3 Sample Analysis
   - 5.4 Instrument Run QC
6. Secondary Data Review
7. Reporting Limits
8. Safety
9. Waste
10. References/Documentation

All procedures must be reviewed by the Laboratory Director and approved by the Technical Director . Copies will be distributed to each work group. Analytical methods must be maintained to insure that they are current with approved regulatory requirements. As changes are made to the regulations, the Laboratory Director will review the procedures for significant changes in methodology and implement changes to current procedures when needed..

7.2 Plans

Microseeps will maintain the quality assurance plan and other written plans as required. Additional plans may include safety, waste management, and project work plans.

7.2.1 Project Work Plans

7.2.1.1 Quality Assurance Project Plans

A Quality Assurance Project Plan (QAPP) describes the policies, organization, objectives, functional activities, and specific quality assurance/quality control (QA/QC) procedures that will be used to achieve the data quality goals and objectives associated with samples collected from a specific site.

Quality Assurance Project Plans that are contractual requirements for a particular job will be prepared at the direction of the Laboratory Director in accordance with "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS 005/80, U.S. Environmental Protection Agency (USEPA), 1983.

When the client has prepared a Quality Assurance Project Plan and made it available to the laboratory, these plans will be collected and reviewed by the laboratory director. It is the client's responsibility to inform and document any exceptions to approved analytical procedures. It is the responsibility of the laboratory director to document, in the form of an internal project plan, any exceptions to Microseeps SOP's and to distribute the internal project plan to the appropriate laboratory personnel. Other project specific information is provided in the laboratory chain of custody forms and all standard, analysis specific QA/QC requirements, are supplied in the appropriate SOP.

7.2.1.2 Non-Routine Project Work Plans

Contractual project plans that have requirements that vary from routine procedures will be prepared at the direction of the Laboratory Director in conjunction with the Technical Director. Specific information relating to the project must be communicated to the Laboratory prior to the development of the work plan, including all QA/QC requirements.

Project work plans will be prepared at the direction of the Laboratory Director. Project plans are used to indicate specific contractual requirements for individual jobs whenever requirements vary from routine procedures. The extent of these differences will determine the need to prepare a project work plan.

Project plans should include the following information:

Client information - including name, address, and point of contact
Scope - including the number and type of analyses, schedule, quality control requirements and reporting requirements
Distribution
Approval signatures

A plan is reviewed by the Laboratory Director, at a minimum. Plans are distributed to each work group affected. Additional copies may be assigned to specific employees as necessary. Revisions are made to plans as necessary to keep them current. If a plan is no longer relevant, it is recalled by the Laboratory Director.

7.3 Distribution and Revision Control

The top right-hand corner of each page of every plan or procedure will list the section number, revision number, effective date, and page __ of ___.

The first time that a plan or procedure is written, it is revision 0. Once the plan or procedure is approved it becomes revision 1. Subsequent revisions are numbered sequentially beginning with 1.1 for minor changes and whole numbers if significant changes are made. Revisions must be approved and distributed following the same procedure as the original. Revisions are denoted in the document by drawing a line in the margin beside the section(s) containing a revision. A summary of the significant changes is maintained. Distribution and revision control of documents are detailed in Microseeps SOP for Document Control, SOP-ADM5 .

The Quality Assurance/Quality Control Department will maintain the original copy of plans and procedures as well as revisions. In addition, the QA/QC Department will maintain a list of all controlled copy-holders. Controlled copy-holders must acknowledge receipt of every controlled copy in writing. The Technical Director will collect and destroy obsolete documents.

8.0 OBJECTIVE FOR PRECISION, ACCURACY, COMPLETENESS, REPRESENTATIVENESS, AND COMPARABILITY

The EPA defines data quality in terms of precision, accuracy, completeness, representativeness, and comparability. This section defines Microseeps's approach to setting objectives for these data quality characteristics.

8.1 Precision

Precision is a measure of the reproducibility of a set of measurements under a given set of conditions. Precision of laboratory data is determined through duplicate analyses of sample or matrix spikes and is calculated as either the range or relative percent difference (RPD) of the measurements. Range is routinely used to quantitate the precision when measurements are made at or near the reporting limit, while RPD is used for higher levels. If one or both measurements are less than the reporting limit, precision is not calculated.

Data quality objectives (DQOs) for precision may be based on statistical evaluation of laboratory data or determined from EPA methods. DQOs vary from parameter to parameter. If the DQO for precision is not met, corrective action must be performed prior to reporting data.

8.2 Accuracy

Accuracy is the measurement of agreement between a measurement and the true value. It is calculated as the percent recovery or error of standards and spikes.

Accuracy of instrumental measurements is measured using initial and continuing calibration standards. DQOs are established using statistical evaluations of laboratory data or based on EPA guidelines. If a DQO is not met, corrective action must be performed prior to reporting data.

Method blanks and laboratory control standards are used to measure the accuracy of the entire analytical procedure. These samples are carried through the entire procedure from sample preparation to analysis. In addition, surrogates are added to every blank, standard, and sample for chromatographic analyses. DQOs are established based on statistical evaluation of laboratory data of EPA methods. If a DQO is not met, corrective action must be performed prior to reporting data.

Matrix spikes are used to measure the accuracy of the analytical system within a specific matrix. In general, matrix spikes are analyzed at a frequency of 1 in 10 or 20 samples of similar matrix, whenever stable reference materials exist. Matrix spike recoveries are reported for informational purposes only. Laboratory control standard data are used to calculate control limits. Whenever precision, calibration, and system quality control checks are acceptable, large or small matrix spike recoveries are assumed to be due to

matrix effects.

8.3 Completeness

Completeness is a measure of the amount of valid data obtained compared to the amount expected to be generated under normal conditions. Valid data are data that meet Microseeps's general quality control requirements or project specific requirements. Completeness is measured through performance and system audits.

8.4 Representativeness

The extent to which data accurately and precisely reflect the sampling points or environmental conditions is representativeness. Numerous items throughout sampling and sample handling must be controlled to maximize representativeness. These include sample collection, preservation, and holding times. Since Microseeps does not perform sample collection, Microseeps cannot accept responsibility for representativeness of sample collection.

At the laboratory, samples are homogenized by stirring, shaking, crushing, or blending, as appropriate to enhance the representativeness of the aliquots used for analysis. If the sample cannot be homogenized, Microseeps will discuss the specifics with the client and proceed according to a mutually agreed upon course of action.

8.5 Comparability

Comparability is the confidence level with which one set of data can be compared to a related set of data. Microseeps uses EPA-approved methodology, whenever available, participates in internal and external performance evaluation programs, and uses standard reference materials for sample analysis as means of enhancing comparability.

9.0 INTERNAL QUALITY CONTROL CHECKS

To determine that laboratory procedures are under control, a variety of quality control samples are analyzed. Included in these QC samples are blanks, duplicates, matrix spikes/matrix spike duplicates, initial and continuing calibration verification standards, and laboratory control standards.

9.1 Blanks

There are different types of blanks that can be used to monitor various phases of the analytical process. The types of blanks are trip, field, equipment, method (preparation), and calibration blanks. Trip, equipment, and field blanks are used to assess sample collection procedures and are treated by the laboratory as ordinary samples. They are not part of the laboratory's internal QC and will not be discussed here.

9.1.1 Method (Preparation) Blanks

Method blanks are reagent water or, for solid/waste matrices, sand or an appropriate solvent is carried through the entire analytical process to monitor contamination introduced during processing. Method blanks are prepared with every batch of samples or 1 in every 20 samples, whichever is more frequent. For organic analyses, surrogates and internal standards are added to the method blank.

Method blanks are analyzed prior to sample analysis. If the analytes of interest are below the laboratory's quantitation limit, sample determinations can proceed. If analyte concentrations are found above the quantitation limits, the source of the contamination must be identified and corrected. The reagents used for sample preparation must be checked for contamination and the samples associated with the method blank must be reprepared.

9.1.2 Calibration Blank

A calibration blank is an appropriate solvent for the method that is analyzed at the point if instrumental measurement to establish and monitor the baseline. Calibration blanks are analyzed after each initial and continuing calibration verification standard. The result must be below the laboratory's quantitation limit for analysis to proceed. If the result of the analysis is above the quantitation limit, the source of contamination must be identified and eliminated. The one exception involves the presence of common laboratory solvents as defined by the EPA.

9.2 Duplicates

Duplicates are analyzed at a minimum frequency of 1 in 10 or 20 samples to assess precision of the analytical procedure. Samples for duplicate analyses are selected at random excluding field and trip blanks, if the laboratory is able to identify these samples.

Precision of the analyses may vary due to the matrix effects of the sample. If the precision is outside established control limits, the duplicate analysis is repeated. If the precision is still outside established control limits and all other QC checks are within control, a matrix effect is assumed.

9.3 Matrix Spike/Matrix Spike Duplicates

Matrix spike analyses may be used as a secondary check of the accuracy of the analytical procedure. Spiked samples are also used to determine matrix effects that may inhibit the laboratory's ability to adequately recover analytes of interest.

One sample per batch or 1 in 10 or 20 samples will be selected for spike analyses. Samples will be selected at random, excluding field and trip blanks, if these samples can be identified by laboratory personnel.

Matrix spike data is provided for informational purposes only. Recovery data is highly dependent upon matrix effects. If acceptable recoveries are observed, it is assumed that sample preparation and analysis have been performed correctly. Because samples are spiked prior to analysis, the concentration of the analyte of interest in the sample may be so high that the spike amount is insignificant. In these cases, spike recovery is meaningless and is not calculated.

9.4 Laboratory Control Standards

Laboratory control standards are known reference materials, independent of the calibration standards that are carried through the entire analytical process. These standards are used to assess the accuracy of the analytical process. Acceptance limits are statistically based upon actual laboratory data. If results are outside acceptance limits, corrective action must be performed before sample analyses can proceed.

10.0 DATA HANDLING

The analytical results produced by the laboratory are the product that the laboratory provides. It is not only necessary to produce precise and accurate data, but to maintain records that enable the laboratory to recreate the conditions under which data are produced. This section defines data collection, record keeping, data reduction, data validation, and reporting procedures.

10.1 Data Collection

All employees are responsible for maintaining laboratory records and documenting them in sufficient detail to recreate the analyses. Manually entered records must be made using permanent ink. Corrections must be indicated by drawing a single line through the incorrect entry, dating and initialing the correction, and coding the reason for the correction. The use of correction fluid or tape, erasure, or other means of making corrections is prohibited. The Standard Operating Procedure SOP-ADM13 defines in more detail the various methods of collecting and recording laboratory data.

The following information, at a minimum, must be recorded:

Method performed listed by Microseeps test code.

Date and time of the analysis and the analyst signature or initials on computer printouts. If an analysis extends over more than one shift or day, each person responsible for part of the analysis must record the date and time their portion of the analysis was initiated.

Instrument

Analytical sequence consisting of a chronological listing of the processing for each standard, quality control check, and sample recorded in an analytical sequence or run log. The laboratory sample number and file name, at a minimum, must be recorded in the log. If the analytical instrument or data system printout does not include the following information, it should also be recorded.

-Client identification, if CLP sample

-QC sample type (i.e., duplicate, preparation blank, etc.) if a QC sample

-Standard identification and volume used for all calibration standards and QC checks

-Dilutions including actual initial and final volumes

-Sample aliquot and final volume

-Instrument reading

-Final results with units

-Percent recovery, RPD, range, or percent difference for all quality control checks

-Units for all variables must be specified.

-Instrument identification and settings must be identified. Settings may be referenced to previous documentation of instrument parameters

-The calibration curve used for quantitation is noted by run date unless included as part of the analytical sequence

-Notes describing any unusual observances

10.2 Reagent and Standard Preparation and Documentation

Chemical reagents and solvents are available in a wide variety of grades of purity, ranging from technical grade to various ultra-pure grades. The purity of the materials required in analytical chemistry varies with the type of analysis, the parameter being measured, and the sensitivity of the detection system. In general analytical reagent grade is satisfactory for most inorganic analyses. Other analyses, such as trace organic often may require special ultra-pure reagents. In cases were the method does not specify the purity of the reagent, it is intended that analytical reagent grade be used. In any case the labels on the container should be checked and the contents examined to verify that the purity of the reagents meets the needs of the particular method involved.

Reagents are always prepared and standardized with the utmost of care against reliable primary standards. They are re-standardized or prepared fresh as often as required by their stability. Stock and working standard solutions are checked regularly for signs of deterioration. Standard solutions are properly labeled as to compound, concentration, solvent, date, name of analyst, and expiration date.

The analyst must properly store reagents and solvents to prevent contamination and deterioration prior to their use. It is recommended that most standard solutions be stored in borosilicate glass bottles with chemically inert stoppers. Plastic containers are recommended for alkaline solutions. Plastic containers must not be used for reagents or solvents intended for organic analyses. It is important that all containers be properly cleaned and stored prior to use. Standard reagents and solvents must always be stored according to the manufacturer's directions. Reagents or solvents that are sensitive to the

light should be stored in dark bottles and in a cool, dark place.

10.2.1 Standards Preparation Logbook

The compound or element name and/or formula are documented along with the final concentration or normality. Also included are the manufacturer's name, lot number, preparation date, analyst name, expiration date, and a brief description. The description of how reagents and standards are prepared may be referenced from a previous description if the exact procedure is used.

10.2.2 Standards Coding/Identification System

Every standard is to be identified by the Group ID code and Logbook Volume Number followed by a dash, Page Number followed by a dash, and Line number of the logbook.

Example if Cyanide spike solution was being entered into Volume 1 of a standards logbook on page 21 as the third entry on that page, the spike solution would be identified as WC1-21-3. (WC identifies the Wet Chemistry Group).

10.2.3 Standard Preparation Procedures

Standard preparation procedures are specific to the analytical determination being made and are defined in detail in the applicable SOP and in the appropriate Standards Logbook.

10.2.4 Reagent Purity

The purity of reagents used in the laboratory is that of reagent grade unless the method specifies a different grade. The required purity for each reagent is specified in the appropriate Standard Operating Procedure.

10.2.5 Traceability

The traceability of each purchased stock standard must be easily accessible. Certificates of analysis of each standard must be maintained in a binder until the standard is depleted or disposed. The certificate is then given to the quality assurance manager for archival.

The traceability of each laboratory prepared standard must be easily traced via standard logbook entries.

10.2.6 Labeling

The labels of reagents and standards purchased by the laboratory, must be identified by the date received, the date of opening, and the initials of the analyst.

Standards prepared in the laboratory must be labeled and identified as described in section 10.2.2.

10.3 Data Reduction

Reducing raw data into a presentable form is the responsibility of the analyst performing the determination. The actual equations used to calculate final results are found in the analytical methods. The following general rounding rules will be used for the calculations.

- Do not round any result until the final answer is obtained.
- To round a figure, determine the number of reportable figures. Look at the digit to the immediate right-most reportable figure. If this figure is greater than 5, round up. If this figure is less than 5, truncate the result after the last reportable figure. If this figure is equal to 5 and there are non-zero digits to the right, round up. If the figure is equal to 5 and there are no non-zero digits to the right, round up when the preceding figure is odd; truncate when the preceding figure is even.

10.4 Data Validation

All data generated by the laboratory will undergo an independent review to ensure compliance with accepted QC standards prior to data entry. The Laboratory Director or experienced analyst will conduct this review. The purpose of this review is to check for precision, accuracy, and completeness. The following items will be verified during this review. Not all items are applicable to each test.

-Have holding times been met?

-Does instrument tune and/or initial calibration meet acceptance limits?

-Have the proper number of continuing calibration/verification standards and blanks been analyzed? Do they meet acceptance criteria?

-Have the proper surrogates and/or spikes been used and are recoveries within control limits

-Is the precision between duplicate analyses within acceptance limits?

If any non-compliant data is encountered during this review, corrective action must be initiated as defined in the SOP-ADM16 Data Validation Procedure.

10.5 Report Preparation

After all of the data has been entered into the LIMS a final report is generated. The final report will be proof read by the laboratory director and/or project manager for completeness and correctness. The following items should be reviewed:

-Client name and address

-Analytical results, units, and reportable figures

-Inter-parametric relationships (i.e., total vs. dissolved, etc.)

-Data reasonableness in respect to sample information

Following the review, if results are acceptable, the report will be signed by the laboratory director and released to the client. A copy of the report will be placed in the client file for storage.

11.0 CORRECTIVE ACTION

An integral part of Microseeps's QA program is the system for identifying, reporting, and correcting deficiencies in the laboratory operation. There are several areas in the laboratory that may require corrective action. It is the responsibility of every employee to be aware of potential problems and to notify the appropriate personnel of situations requiring corrective action.

11.1 Problem Isolation and Identification

Identification and isolation of problems in the laboratory are not always easy tasks. The need to perform corrective action may become apparent at any point of the analytical process. Procedures for some sample handling deficiencies such as improperly preserved sample bottles, broken or missing containers, and holding time violations have been previously addressed.

Corrective action should be initiated as soon as a problem becomes evident. Some situations, such as malfunctioning equipment, are detected by the analyst at the bench. Corrective action for these situations takes the form of repairing the instrument, either internally or through the use of a service call. The corrective action is documented in the instrument maintenance log and the data obtained just prior to the failure is closely scrutinized for acceptability.

Other situations may not be easily identifiable. For example, systematic drift or sensitivity fluctuations may not be identified until the time that data is validated. Other occurrences that may trigger the need for corrective action include the following:

- Recoveries for surrogates, matrix spike, matrix spike duplicates, and laboratory control standards outside of acceptance limits.

- Percent differences for duplicate analyses outside acceptance limits.

- Trends noted in quality control data. For example, 5 or more consecutive surrogate (or other QC check) recoveries below the statistical mean.

11.2 Problem Resolution

Since the need to perform corrective action can be identified at any point, the form of the action varies.

11.2.1 Sample Handling Problems

Problems involving sample handling may include missing or broken containers, discrepancies between the chain of custody and actual shipment, improperly preserved bottles, insufficient volume, and missed holding times. When one of

these problems is identified, the Laboratory Director, Project Manager, or Customer Service Manager is notified. The client is contacted to discuss the possible resolutions to the problem and the resolution is documented. The documentation becomes part of the client's permanent file.

11.2.2 Sample Analysis Problems

Problems incurred during sample analysis bring procedures and data into question. These problems may include the following:

- Unacceptable calibration
- Improper procedures
- Unacceptable blank, LCS, and/or surrogate recovery
- Quantitation error
- Required QC not performed
- Retention time shifts

Resolution is these problems may include re-preparing and reanalyzing samples. Re-calibrating the instrument, and remaking standard solutions and reagents. Standard Operating procedures detail corrective action steps that are specific to an analytical procedure. The documentation becomes part of the client's permanent file.

The person identifying the problem documents the situation and identifies possible sources of the problem. If this individual can immediately correct the situation, for example, by re-calibrating the instrument, they should do so and document the action that was taken. If the problem cannot be corrected immediately, the person should document the situation and notify the Laboratory Director. The Laboratory Director is responsible for ensuring that the appropriate corrective actions are followed.

11.2.3 Performance Evaluation Deficiencies

When the laboratory receives the results for third party performance evaluation studies, the QA Manager starts the investigations into any marginally acceptable and unacceptable results. The investigation includes reviewing the data generated during sample analysis to ensure that a calculation or reporting error has not occurred. If the data appears to meet QC criteria, the next step is to work with the analysts involved to ensure that procedures are being followed properly. The results of these investigations are discussed with the Laboratory and Technical Directors. The Technical Director must approve modifications to analytical methods or support procedures.

11.3 Laboratory Records

A Corrective Action Record (CAR) (Figure 11-1) will be completed for each incident requiring corrective action. The QA Manager will be responsible for maintaining a supply of forms. To track the corrective action, each CAR will be given a unique identifier in the form of YY-XXXX where:

YY = the year in which the CAR was initiated and
XXXX = is the sequential number of the record starting with 0001

The analyst identifying the problem will be responsible for initiating the CAR. They will contact the QA Manager to obtain the next available CAR and complete the top portion of the form. The form will then be forwarded to the Laboratory Director for approval. If necessary the client will be notified of the incident. If the corrective action is acceptable, the Laboratory Director will sign the CAR and forward the notice to the Technical Director. If the Laboratory Director does not agree with the corrective action taken, the notice will be returned to the laboratory staff for additional work. Once the Laboratory Director approves the corrective action, the CAR is signed and forwarded to the Technical Director.

The Technical Director will review the CAR. If the Technical Director concurs with the action taken, the CAR is signed and the issue is closed. If the Technical Director does not agree with the action that has been taken, the CAR is returned to the Laboratory Director and/or analyst for further action.

The QA Manager will initiate CARs in response to third party audits and deficiencies on performance evaluation studies. In the case of third party audits, if the resolution to finding is easily identifiable, the QA Manager will discuss the situation with the Laboratory Director and the corrective action will be implemented. If the finding concerns a procedural change or is interdisciplinary, a task team will be formed to investigate the problem and develop an appropriate course of action. The task team's findings will be presented to management for discussion and possible implementation. Once a corrective action plan is implemented, the situation will be monitored for a reasonable period of time to ensure that the action has been effective.

The Technical Director will prepare a report for management on a monthly basis that summarizes all corrective actions. The report will provide a brief description of the problem, the steps that are being taken to correct the situation, and the status of the item.

Figure 11-1
Corrective Action Report

Sample Number(s):_____ CAR No. _____
Lab Group: _____ Date: _____

NONCONFORMANCE

1. Describe the nonconformance

_____
_____
_____
_____

2. Describe the corrective action taken

_____
_____
_____
_____

Signature of Person Initiating the Nonconformance_____

Date: _____

APPROVALS

Laboratory Director
The client was/was not contacted

Additional action _____is needed
_____is not needed

Signature _____Date_____

QA Manager
Additional action _____is needed
_____ is not needed

Signature: _____Date_____

COMMENTS

_____
_____
_____

12.0 PERFORMANCE AND SYSTEMS AUDITS

This section describes Microseeps's approach to performance and systems audits. Performance evaluation audits enable Microseeps to measure the precision, accuracy, and comparability of laboratory-generated data through the use of blind reference materials. Microseeps participates in performance evaluation studies sponsored by the EPA and several state agencies. Systems audits are conducted to monitor actual laboratory practices against quality control requirements.

12.1 Performance Evaluation Audits

Performance evaluation studies are completed as required to maintain necessary certifications, at a minimum. Additional studies may be run at the Laboratory Director's discretion.

Prior to analysis, the analyst prepares the samples following the instructions provided. If the samples are submitted as a blind study, the Laboratory Director or QA Manager prepares the samples. Samples are analyzed as soon as possible after opening the vials to avoid sample deterioration.

The QA Manager evaluates the results of the Performance Evaluation prior to reporting them to the appropriate agency.

Once the evaluation report from the EPA or appropriate agency is received, the QA Manager summarizes the results. Unacceptable results are investigated to determine the cause of the failures. The following points are addressed during the investigation:

- Potential for reporting/calculation errors
- Preparation of calibration standards
- Evaluation of quality control data associated with the analysis
- Evaluation of analytical technique and instrument performance

Additional blind samples may be submitted for analysis until the Laboratory Director and QA Manager feel that the problems have been corrected.

12.2 System Audits

Systems audits may be conducted internally or by an independent third party.

12.2.1 Internal Audits

The Laboratory Director and QA Manager will develop a schedule to ensure that all laboratory groups and programs are audited each year. The QA Manager will prepare an audit plan. The plan will delineate the activities and records that will be reviewed. The Laboratory Director is consulted to ensure that all areas of concern are addressed.

The audit is performed following the prepared plan. Notes are made based upon observations, interviews, and record reviews. The notes should include specific details of the process. Using the notes, an audit report is prepared. The report will include discussions off strengths and weaknesses that were noted. Whenever deficiencies are noted, specific examples must be included as well as potential explanations and recommendations for improvement. A copy of the audit report is forwarded to the Laboratory Director for review and discussion. If deficiencies are noted, follow-up is conducted to monitor the effectiveness of corrective action.

12.2.2 External Audits

The Technical Director acts as the liaison between Microseeps and the third party auditor. The Technical Director is responsible for notifying the laboratory staff of upcoming audits. This notification will include the agency that will perform the audit, the reason for the audit, the dates involved, and the areas of concern. During the audit, laboratory personnel will be called upon as necessary. The Technical Director will prepare a report summarizing the findings of the post audit meeting. Following review of this report, the Laboratory Director, QA Manager, and additional laboratory personnel, as necessary, will prepare a response. Follow-up will be performed as necessary.

13.0 QA REPORTS TO MANAGEMENT

Regular discussions of quality assurance issues are necessary to provide a forum in which upper management are informed of problems and changes that affect the laboratory operations.

13.1 Meetings

Each quarter, the QA Manager will meet with the Laboratory Director and President to discuss quality issues. This meeting will serve as a general discussion of factors affecting quality and will include the following topics at a minimum:

- personnel changes
- instrument changes
- audit findings
- certification changes

At the end of the meeting, the QA Manager will prepare a summary of the discussion and set an agenda for additional follow-up, if required.

ANALYTICAL METHOD

AM15.01

MICROSEEPS

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Pittsburgh, PA 15238
(412) 826-5245

MICROSEEPS

ANALYTICAL METHOD AM15.01

ANALYSIS OF DISSOLVED GASES IN WATER

1.0 Scope and Application

1.1 Method AM15.01 may be used to determine the concentration of dissolved gases in water samples. Specifically, Method AM15.01 is used to determine the dissolved concentration of the permanent gases:

methane
oxygen
nitrogen
carbon dioxide
carbon monoxide

1.2 This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Summary of Method

2.1 Analysis of the permanent gases in a water sample is accomplished by transferring 30 ml of the sample plus 10cc of helium into a 50cc gas tight syringe. After equilibration, the headspace gases are analyzed with a gas chromatograph, using a series-bypass valve configuration and a thermal conductivity detector (TCD). The sample (and standard calibration gas) is introduced into the columns by the mechanical injection of a sample loop. The data is transferred to a microcomputer where it is converted to digital format, stored, and processed using a chromatography data system (Chrom Perfect Direct, Justice Innovations).

3.0 Interferences

3.1 The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air will seriously skew the results. The analyst must take great care to ensure that air is flushed from the 50cc gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

ANALYTICAL METHOD

15.02

MICROSEEPS

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MICROSEEPS

ANALYTICAL METHOD AM15.02

ANALYSIS OF DISSOLVED GASES IN WATER

1.0 Scope and Application

1.1 Method AM15.01 may be used to determine the concentration of dissolved gases in water samples. Specifically, Method AM15.01 is used to determine the dissolved concentration of the permanent gases:

methane
oxygen
nitrogen
carbon dioxide
carbon monoxide

1.2 This method is recommended for use by, or under the supervision of, analysts experienced in sample preparation, the operation of gas chromatographs and in the interpretation of chromatograms.

2.0 Summary of Method

2.1 Analysis of the permanent gases in a water sample is accomplished by transferring 30 ml of the sample plus 10cc of helium into a 50cc gas tight syringe. After equilibration, the headspace gases are analyzed with a gas chromatograph, using a series-bypass valve configuration and a thermal conductivity detector (TCD). The sample (and standard calibration gas) is introduced into the columns by the mechanical injection of a sample loop. The data is transferred to a microcomputer where it is converted to digital format, stored, and processed using a chromatography data system (Chrom Perfect Direct, Justice Innovations).

3.0 Interferences

3.1 The most likely source of "interference" is ambient air. Due to the relatively high concentrations of oxygen and nitrogen, a very small amount of air will seriously skew the results. The analyst must take great care to ensure that air is flushed from the 50cc gas tight syringe before sample preparation and that no air has entered the syringe or needle prior to injection of the sample into the gas chromatograph.

3.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. An unrestricted flow of pure helium from a 10 psig source should be allowed to flow through the sample loop for 30 seconds prior to each analyses.

3.3 The analyst should demonstrate the absence of carryover contamination by analysis of the contents of the sample loop when purged with helium. This demonstration should be performed prior to the analysis of a sample set and when carryover contamination is suspected (after high samples). In the event that `ghost peaks' (peaks similar to previous sample) appear when a pure helium sample is analyzed, measures should be taken to eliminate the carryover contamination.

3.4 Extra peaks in a chromatogram can be actual peaks from a previous run. Contamination from late eluting peaks can occur when the time between successive injections is too short.

3.5 The analyst should be certain that all peaks have eluted from the previous analysis prior to analyzing any sample or standard. If samples or standard chromatograms contain suspected `extra peaks' the sample should again be analyzed after a clean baseline is established.

4.0 Apparatus and Materials

4.1 Sample vials: 40 ml VOA glass vials (QEC #2112-40ml or equivalent). Vials should be free of all hydrocarbons and compounds of interest prior to use.

4.2 Septa: Foil faced silicon (Integrated Liner Technologies, Inc.).

4.3 Syringe: Hamilton 50cc locking gas tight (#1050TLL or equivalent).

4.4 Gas Chromatograph: The chromatograph is a Gow Mac 580 equipped with a thermal conductivity detector, sample loop, gas sampling valve, series-bypass valve, 1/8 inch x 8 foot Porapak Q column, and a 1/8 inch x 8 foot molecular sieve 5A column. This arrangement allows rapid turn-around for consecutive analyses and a clean baseline for accurate, reproducible results.

4.5 Data Collection: The output of the chromatograph is directed to a microcomputer where the signal is converted to digital format, stored, and processed using a chromatography data system (Chrom Perfect Direct, Justice Innovations, Palo Alto, CA).

4.6 Automated valve control: Digital control is provided by the microcomputer though the Chrom Perfect Direct software. This control provides constant start and stop times for directing carrier gas flow though the sample loop and for activating/deactivating the series/bypass valve. The event times are programmed and saved using the method editor module of Chrom Perfect.

5.0 Sample Preparation and Analysis

5.1 Remove the sample (VOA) vials from the refrigerator. Let the samples reach ambient temperature over a period of 4 hours.

5.2 Using a clean 50ml gas tight, locking syringe withdraw 30ml of water from the bottom of the sample vial.

5.3 Withdraw 10cc of helium from a reservoir and lock the syringe.

5.4 Shake the syringe by hand (or use a wrist action shaker) for five minutes.

5.5 With the syringe in a vertical position, slowly inject the 10cc of headspace gas into the gas chromatograph sample loop though a septum fitting. The sample loop should be switched into the carrier gas flow stream (ten port valve activated) immediately after the sample loop has been filled with sample at atmospheric pressure. The flow though the sample loop is monitored by a flow meter connected to the sample loop vent port on the gas chromatograph.

6.0 Calibration and Results

6.1 The standard calibration gas should be introduced in the same manner as described in section 5.5 above. Measured peak areas are converted to concentrations in percent by volume using certified commercial gas standards traceable to NIST standards. (Standard Mix 237, Scott Specialty Gases). Dilutes may be made to achieve multi point calibration curves.

6.2 At the beginning of a project or sample set, standards of appropriate calibration ranges will be run at least three times or until the results agree with a percent standard deviation no greater than 10%.

6.3 The instrument response (for any one subsequent standard in section 6.1 above) must not vary by more than 20%.

7.0 Quality Control

7.1 If the parameters set forth in section 6.3 are not met, the analytical program will be terminated until the cause is determined and a solution is effected.

7.2 The analyst should demonstrate the absence of ambient air in the sample preparation system by filling a sample syringe with helium and injecting 10cc of helium into the sample loop in the same manner as a sample. The results of this 'syringe blank' should show oxygen and nitrogen levels below the minimum detection levels.

7.3 Before and during sample analysis, instrument blanks (sample loop filled with flush helium) should be analyzed to assure the absence of interferences as described in section 3.0 above.

7.4 Standards analyzed during the course of analyzing samples may be averaged into the calibration table as well as being used for peak identification. All chromatograms should be examined by an experienced analyst.

7.5 Throughout analysis the gas samples are injected mechanically utilizing a sample loop to achieve a uniform sample size from a flow directly from the sample preparation syringe. The uniform sample size assures consistent and accurate results.

7.6 The water sample is withdrawn from the 40ml VOA vial immediately after opening. The 30ml of sample is withdrawn from the bottom of the 40ml vial and the remaining sample, that which has had contact with room air, is discarded.

7.7 Calibration records are generated and stored. All such records will be maintained in the laboratory during the course of the project and there after as determined by the client.

8.0 Instrument Conditions

8.1 Gas Chromatograph:

Injection Port A Temp. 90 °C
Thermal Conductivity Detector Temp. 90 °C.
Oven Temp. 70 °C. isothermal
Initial T.C.D. Signal Attenuation 1
Carrier Gas Regulator Pressure 80 psig
Sample carrier flow 40 cc/min.
Reference flow 40 cc/min.
Valve Air Pressure 60 psig.